METHOD AND DEVICE FOR DETERMINING THE RESONANT FREQUENCY OF A RESONANT
PIEZOELECTRIC SENSOR

Field of the Invention

The present invention relates to a method and a device for
5   determining the resonant frequency of piezoelectric resonant sensors
subject to a load.

Background of the Invention

Piezoelectric resonant sensors based on AT-cut quartz crystals vibrating
in   thickness-shear   mode   (TSM)   are   used   as   quartz-crystal
10 microbalances (QCM), film-thickness monitors, sensors for physical-
chemical properties of fluids, and as transduction devices for
chemical and biochemical sensors.

Quartz crystal resonant sensors are widely used in the chemical,
medical, biotechnology, environmental, food, materials, and process
15 control fields.

The primary output signal of this kind of sensors is the crystal resonant
frequency, which needs to be accurately determined since it directly
relates to the quantity to be measured.

To this purpose, oscillator circuits are typically used in which the crystal
20 is inserted as the frequency-controlling element. However, when the
sensor is subject to heavy acoustic and/or dielectric loads, such for
measurements in contact with liquids or with viscoelastic media, the
sensor resonant frequency and the output frequency of the oscillator
circuit can become significantly different, thereby resulting in large
25 inaccuracies and performance degradation.

As a limiting case, under high damping, the oscillator may stop
operating properly and cease to sustain oscillations, therefore
restricting the sensor operating range.

Such a situation can be represented by an extension of the equivalent

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circuit known in the literature as the BVD (Butterworth-Van Dyke) model, in which the characteristic parameters of a sensor subject to a load include a first capacitance representing the electrical behavior of the sensor associated to the intrinsic capacitance of the crystal,

5   and a second capacitance resulting from contact with, or immersion into, a liquid whose conductance is assumed to be negligible. Both such capacitances in the extended BVD model are connected in parallel and they together form the so-called sensor parallel, or static, capacitance.

10 The parallel capacitance, whose effect combines to the increase of dissipation losses caused by the load, is the parameter that negatively affects the determination of the quantity to be measured, that is the sensor resonant frequency, and therefore the parallel capacitance. has to be compensated for improving the measurement accuracy.

15 Oscillator circuits are already known in the literature that introduce the compensation of the sensor parallel capacitance. However, in such known oscillator circuits the compensation implies a manual adjustment which is time consuming and error prone. Moreover, such known oscillator circuits do not allow to measure the value of the

20 compensated capacitance, which, on the contrary, can be of significant interest in several applications.
Examples of studies on the compensation of the sensor parallel capacitance have been carried out by the inventors themselves. Reference can be made, for instance, to "Oscillator Circuit

25 Configuration for Quartz-Crystal-Resonator Sensors Subject to Heavy Acoustic Load, Electronics Letters, 36, 7 (2000); "Improving the Accuracy and Operating Range of Quartz Microbalance Sensors by a purposely Designed Oscillator Circuit" - IEEE TRANS. ON INSTR. AND MEASUR., Vol. 50, no. 5, October 2001; and "Accuracy and Range

Limitations in Oscillator-Driven QCM Sensors" - Proceedings of 7th National Conference on Sensors and Microsystems, Bologna, 4-6 February 2002.

In such studies, an oscillator circuit was proposed which includes a

5   section working as a negative capacitance that is summed to the sensor parallel capacitance, thereby removing the perturbing effects that prevent the accurate measurement of the sensor resonant frequency.

This approach, though provides satisfying results, brings about some

10   limitations. In fact, the particular configuration adopted to simulate the negative capacitance in some of the above referenced studies can become unstable under certain circumstances; moreover, in none of the circuits of the above referenced studies the sensor has one terminal connected to ground, which instead would be desirable

15   in several electrochemical and biological applications.

A different approach to compensate for the parallel capacitance has been proposed by A. Arnau *et al.* ("Circuit for continuous motional series resonant frequency and motional resistance monitoring of quartz crystal resonators by parallel capacitance compensation" REV.

20   OF SCIENT. INSTR. - Vol. 73, no. 7 - July 2002) introducing a circuit which, however, requires a number of lengthy calibration operations to be performed with the unperturbed sensor. A solution is also mentioned which would enable the automation of the calibration steps of the system and the parallel capacitance compensation, but this would

25   make the circuit even more complex and costly.

Anyway, none of the oscillator configurations so far proposed has made it possible the determination of the instantaneous value of the compensated capacitance, which is an additional and very important parameter.

As an alternative to the use of oscillator circuits, the impedance spectrum of the sensor can be measured by means of impedance analyzers. They, however, are costly instruments, require specialized personnel to operate them, and, as such, are essentially limited to
5   laboratory use.

Summary of the Invention

The task of the present invention is to propose a method and a device that offer the typical advantages provided by the oscillators in terms of compactness, ease of use for unspecialized personnel, and low
10 cost, while, at the same time, overcoming the limitations of the systems known to date.

Within the scope of this task, one object of the present invention is to propose a method which allows to determine with very high accuracy the value of the resonant frequency of a resonant sensor subject to an
15 acoustic and/or dielectric load.

Another object of the present invention is to propose a method of the above cited type which enables to take extremely accurate measurements even in the cases where the resonant sensor is subject to high damping.

20 A further object of the present invention is to propose a method of the above cited type which enables to make the compensation of the sensor parallel capacitance of a resonant sensor automatic.

Still another object of the present invention is to indicate a device which is low-cost and easy to implement and use as to determine in a
25 fully automated way the value of the resonant frequency of a resonant sensor subject to an acousto-mechanical and/or dielectric load.

These objects are achieved by the present invention, which relates to a method for determining the value of the resonant frequency of a

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loaded resonant sensor in accordance with claim 1.

In order to obtain a high measurement accuracy, the proposed technique to compensate the sensor capacitance introduces the fundamental innovation of being completely automatic, without any
5   adjustment required to the user. Such a technique is based on the simultaneous and independent excitation of the sensor at two different frequencies, and on the use of two separate feedback loops. Preferably, one first frequency is the series resonant frequency of the sensor, while the second frequency is lower than the series resonant
10 frequency of the sensor.

The invention further relates to a device for determining the value of the resonant frequency of a resonant sensor subject to a load, in accordance to claim 11.

The oscillator circuit of the device according to the present invention
15 proposes and implements a technique to obtain an active and automatic compensation of the sensor parallel capacitance, and to maintain the oscillation frequency of the circuit constantly equal to the frequency where the phase of the sensor impedance is null.

Under the condition of neutralization of the parallel capacitance, such
20 a frequency exactly corresponds to the sensor resonant frequency, irrespective of the degree of damping. Moreover, the circuit automatically follows the above frequency, thereby providing an accurate and reliable measurement of the sensor response.

The excitation at the lower frequency and the first feedback loop
25 enable to detect the sensor response due to the parallel capacitance only. By properly processing such a response, the automatic cancellation of the parallel capacitance is performed. In this condition, the second feedback loop, which is a phase-locked loop (PLL), allows to keep the higher frequency constantly locked to the

sensor resonant frequency.

In addition to the instantaneous values of the sensor resonant frequency and damping, the circuit advantageously provides an output parameter related to the value of the compensated
5   capacitance.

Thanks to the characteristics of the method and the device according to the present invention, it is possible to continuously measure the evolution of the parallel capacitance in case of changes of it during the course of a measuring experiment. This may be particularly
10 important in specific applications.

Brief Description of the Drawings

Further characteristics and advantages of the present invention will be more clear by the following description, made with illustrative and not limiting purposes, with reference to the attached schematic drawings,
15 in which:

-   Figure 1 is a detailed electrical diagram representing the equivalent circuit of a quartz crystal resonant sensor subject to acousto-mechanical and dielectric loading;

-   Figure 2 is a simplified electrical diagram of the same equivalent
20   circuit of Figure 1; and

-   Figure 3 is an electrical block diagram of a device according to a possible embodiment of the present invention.

Modes for Carrying out the Invention

A quartz crystal resonant sensor subject to both acousto-mechanical
25 and dielectric loading can be represented around its fundamental resonant frequency by the equivalent circuit of Figure 1 (extended BVD model).

In the circuit, the components L_1, C_1 and R_1 form the mechanical (i.e. motional) branch of the model and represent the equivalents of mass,

elastic compliance, and mechanical losses, respectively, of the unloaded sensor. The capacitor C_0 represents the dielectric behavior of the sensor associated to the crystal capacitance.

The acousto-mechanical load is represented by the equivalent impedance Z_{Leq}, while C_P is the additional capacitance arising from contact with, or immersion into, a liquid whose conductivity is assumed to be negligible.

More specifically, Z_{Leq} can be purely inductive in the case of simple mass accumulation, or complex when an appreciable damping is also present, such as for instance in case of dense and viscous liquids or with viscoelastic films placed on the sensor.

The quantity of primary interest because it directly relates to the load and it is not influenced by stray capacitances in parallel to the sensor, is the series resonant frequency f_s given by:

$$f_s = \frac{1}{2\pi\sqrt{L_T C_T}} \tag{1}$$

where L_T and C_T respectively represent the total, i.e. inclusive of the load, inductance and capacitance in the motional branch of the sensor equivalent circuit, as resulting from the simplified electric diagram of the equivalent circuit shown in Figure 2.

In this diagram, the parallel, or static, capacitance is indicated by the capacitor C_0^*, and its value is given by the sum of the capacitances C_P and C_0 shown in Figure 1, that is to say $C_0^* = C_0 + C_P$.

Typical values of f_s are in the order of 5-30 MHz, depending on the thickness of the particular sensor used.

In order to determine the amount of dissipation losses at the frequency f_s caused by the total resistance R_T, it is also useful to measure the degree of damping or, equivalently, the quality factor Q given by:

$$Q = \frac{2\pi f_s L_T}{R_T} = \frac{1}{2\pi f_s C_T R_T}$$ (2)

The block diagram of a device according to a possible embodiment of the present invention is shown in Figure 3.

The sensor, represented by its equivalent circuit inclusive of the load

5   (Figure 2), is included within the dashed frame S.

The block named C_C represents a variable capacitance whose value is controlled by the voltage V_C. To implement such a variable capacitance, a fixed capacitance connected in series to the output of a voltage amplifier with a voltage-controlled gain can be used for

10   instance. As an alternative, other known circuit schemes can be used, including for instance a varactor (or varicap) diode, or any devices and configurations able to provide a voltage-controlled variable capacitance.

The voltage waveform V_{HL} is the sum of the sinusoidal signal V_L having

15   a preset frequency f_L generated by the oscillator OSC, and of the sinusoidal signal V_H having a frequency f_H generated by the voltage-controlled oscillator VCO.

The frequency f_H of the signal V_H is taken as the output frequency f_{out} of the whole oscillator circuit, and it will be shown below that it is

20   constantly maintained equal to the sensor series resonant frequency f_s. Preferably, the frequency f_L is lower than f_H. For example, in the experimental tests performed with 10-MHz resonant sensors (f_H = 10 MHz), the frequency f_L was set to 50 kHz. Other values of f_L can be used as well, provided that they are suitably lower than the frequency

25   f_H to make the discrimination between such two frequencies effective, and therefore make the following considerations valid.

Assuming to avoid the use of particularly selective filters, which tend to

be complex and costly, the upper limit for f_L can be reasonably set to a couple of decades lower than the sensor resonant frequency.

As far as the basic principle of the proposed method is concerned, the frequency f_L might be as well chosen of a suitably larger value than the sensor resonant frequency. However, such a choice would cause practical problems related to the need for operating part of the circuit at very high frequency (in the order of tens or hundreds megahertz), which would in turn introduce critical issues that are instead avoided by the adopted choice.

In the frequency domain, the differential voltage ($V_2 - V_1$) is related to the voltage V_{HL} through the following expression:

$$V_2 - V_1 = V_{HL} Z_4 \alpha \left[Y_T + j\omega C_0^* - j\omega C_{C\alpha} \right] \qquad (3)$$

where:

- $C_{C\alpha} = \left[1/\alpha - 1 \right] C_C$

- $Z_4 = \dfrac{R_4}{1 + j\omega R_4 C_4}$

- $\alpha = \dfrac{R_3}{R_2 + R_3}$

- $Y_T = \left[j\omega L_T + R_T + \dfrac{1}{j\omega C_T} \right]^{-1}$

The values of R_4 and C_4 are properly chosen so that the impedance Z_4 be dominated by R_4 at the lower frequency f_L, and by C_4 at the higher frequency f_H.

The expression (3) simplifies in two different expressions when it is considered at either the lower frequency f_L or the higher frequency f_H.

At frequency f_L the sensor is far from the resonance and its equivalent circuit reduces to the parallel capacitance C_0^*. The expression (3) therefore becomes:

$$V_2 - V_1 = j\omega V_L R_4 \alpha \left(C_0^* - C_{C\alpha} \right) \qquad (4)$$

The expression (4) shows that, by adjusting the compensating capacitance C_C, it is possible to reach the condition where the capacitance C_0^* is neutralized by $C_{C\alpha}$ by means of the detection of the situation where the differential voltage ($V_2 - V_1$) is zero.

This is performed in an automatic and continuous way by the part of the circuit that uses the blocks PB, AD2, SF, M2, I2, and C_C, to implement a feedback loop that keeps locked to the condition ($V_2 - V_1$)=0.

In fact, the low-pass filter PB extracts from the signal ($V_2 - V_1$) the component at the low frequency f_L corresponding to the expression (4). Such a component of the signal ($V_2 - V_1$) is amplified by the differential amplifier AD2. The 90° phase shifter SF and the analog multiplier M2 perform a synchronous detection of the component of ($V_2 - V_1$) at the frequency f_L, and transform the component in quadrature with respect to V_L into a DC voltage.

The integrator I2 forces the output of the analog multiplier M2 to zero, thereby constantly nulling the static error in the loop. The DC output voltage V_C of the integrator I2 adjusts the variable capacitance C_C.

In this way, the sensor parallel capacitance C_0^* is automatically and constantly compensated by the capacitance $C_{C\alpha}$. The DC voltage V_C is taken as an additional output to provide an instantaneous value of the compensated parallel capacitance C_0^*.

At the frequency f_H, thanks to the above described method of

automatic compensation of the capacitance C_0^*, the expression (3) becomes:

$$V_2 - V_1 = \frac{\alpha V_H}{j\omega C_4} \left(j\omega L_T + R_T + \frac{1}{j\omega C_T} \right)^{-1} \qquad (5)$$

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The multiplier M1, integrator I1, and voltage-controlled oscillator VCO together form a phase-locked loop (PLL) feedback system.

In fact, the multiplier M1 transforms the component of the differential
10   voltage $(V_2 - V_1)$ in quadrature with respect to V_H at the frequency f_H into a DC voltage that is constantly forced to zero by the integrator I1.

To occur this condition, the output frequency f_h of the oscillator VCO is kept necessarily to the frequency at which the conductance Y_T of the sensor is purely real. Such frequency is the same of the series resonant
15   frequency f_s.

Therefore, the output frequency f_{out}, that is f_H, is constantly equal to f_s, irrespective of the loading conditions.

The high-pass filter PA, differential amplifier AD1, and peak rectifier RP form a circuit section dedicated to the measurement of the sensor
20   dissipation at resonance.

In fact, the high-pass filter PA extracts from the signal $(V_2 - V_1)$ the component at the higher frequency f_H, which by means of the two above described feedback loops is constantly kept equal to the resonant frequency f_s. Therefore, the amplitude of such a component of the signal $(V_2 - V_1)$ is proportional to the term $1/R_T$ or, equivalently, to
25   the quality factor Q of the sensor, as expressed by equation (2). The peak rectifier RP then provides a DC voltage proportional to $1/R_T$ that is taken as a further additional output of the whole circuit.

EXPERIMENTAL RESULTS

A prototype of a device including an oscillator based on the electric diagram of Figure 3 was assembled using commercially-available components selected among those having proper characteristics. In the prototype, the variable capacitance was implemented as described above, that is by means of a fixed capacitance connected in series to the output of a voltage amplifier with a voltage-controlled gain.

As the piezoelectric resonant sensors, 10-MHz AT-cut TSM quartz crystals were used. The frequency of the signal V_L was set to 50 kHz.

The sensors were immersed into four liquids determining different loading conditions, namely acetone, trichloroethylene, ethanol, and ethylene glycol.

As a preliminary step, the differences between the exact unloaded series resonant frequency f_s of the sensors, nominally equal to 10 MHz, and the corresponding frequency values in each of the four loading conditions were measured by means of an impedance analyzer and the obtained results were considered as the reference values (Δf_s reference in Hz).

Therefore, a second test was performed with the sensors connected to the proposed oscillator circuit in which the parallel capacitance compensation section was disabled, and the oscillator output frequency was measured in the different loading conditions to determine the values of the frequency shift Δf_s with respect to the unloaded case, and in a third test the oscillator output frequency was measured in the different loading conditions with the parallel capacitance compensation section enabled, and the values of the frequency shift Δf_s with respect to the unloaded case were determined for comparison. The results of the tests are reported in the following Table 1.

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TABLE 1

	Acetone	Trichloroethylene	Ethanol	Ethylene Glycol
Δf_s reference [Hz]	3210	5304	5462	20838
Δf_s oscillator without the capacitance compensation [Hz]	2750	3366	3531	Circuit unable to sustain oscillations
Δf_s oscillator with the capacitance compensation enabled [Hz]	3232	4942	5375	20529
Relative error of Δf_s without the capacitance compensation	14.3%	36.5%	35.3%	- - -
Relative error of Δf_s with the capacitance compensation enabled	0.7%	6.8%	1.6%	1.5%
Controlling voltage V_C of the compensating capacitance C_C [mV]	365	319	389	518
Value of the compensating capacitance C_C [pF]	9.92	7.81	11.02	16.92

In addition to the percent errors occurred in the second and third tests with the proposed oscillator circuit, Table 1 also reports in the last two rows the measured values of the voltage V_C and the correspondent

values of the compensating capacitance Cc.

The following Table 2 reports the corresponding values between the compensating capacitance Cc and the controlling voltage Vc measured in the circuit.

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TABLE 2

Capacitance C_C [pF]	Voltage V_C [mV]
2.10	191
3.85	231
5.43	268
6.65	295
8.21	330
8.75	342
10.20	375
12.07	417
12.40	420
13.64	455
15.04	485
16.85	527
18.20	555
20.91	604
21.93	641
24.85	707
26.50	743
30.14	828
32.87	887
39.30	994

The experimental results shown in Table 1 demonstrate that the device

according to the invention with the automatic capacitance compensation system enabled provides an accuracy improvement of more than one order of magnitude over the uncompensated case. Moreover, the oscillator operates correctly with high metrological performances even when loaded by ethylene glycol, which is a liquid that, due to the high dielectric constant and induced damping prevents the circuit from sustaining oscillations in the absence of capacitance compensation.

Moreover, the data in the last two rows of Table 1 show that the oscillator, by means of the voltage V_C and the relationship with the compensating capacitance C_C, is capable to determine the value of the compensated parallel capacitance C_0^*. As expected, such a value increases with increasing the dielectric permittivity of the liquid.

The basic principles of the present invention are not limited to the quartz sensors herein described as an example, but they can as well find application with piezoelectric resonant sensors in general.